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Homogeneous flow and size dependent mechanical behavior in highly ductile Zr₆₅Ni₃₅ metallic glass films

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ABSTRACT

Motivated by recent studies demonstrating a high strength - high ductility potential of nano-scale metallic glass samples, the mechanical response of freestanding Zr₆₅Ni₃₅ film with sub-micron thickness has been investigated by combining advanced on-chip tensile testing and electron microscopy. Large deformation up to 15% is found for specimen thicknesses below 500 nm with variations depending on specimen size and frame compliance. The deformation is homogenous until fracture, with no evidence of shear banding. The yield stress is doubled when decreasing the specimen cross-section, reaching ~3 GPa for small cross-sections. The fracture strain variation is related to both the stability of the test device and to the specimen size. The study concludes on clear disconnect between the mechanisms controlling the onset of plasticity and the fracture process.

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1. Introduction

Compared with crystalline metals that show large tensile ductility with significant uniform elongation, bulk metallic glasses (BMGs) are essentially brittle at room temperature [1–3]. Narrow (~10 nm-thick) shear bands develop as the stress gets large enough with severe plastic-strain localization leading to catastrophic failure [1–3]. Recent reports have shown that the brittle-like behavior is mitigated when the sample size is reduced down to the sub-micron scale. This mechanical size effect has been predicted by molecular dynamic simulations [4,5] and experimentally demonstrated using compression (or tension) test of micropillars [6–9] or *in-situ* transmission electron microscopy (TEM) tensile tests [8,10,11].

Volkert et al. [6,12] reported a transition from shear band mediated plastic flow to homogeneous deformation under

compressive loading when the diameter of amorphous Pd₇₇Si₂₃ micropillars is below 400–500 nm. Jang and Greer [7] and Chen et al. [13] confirmed this trend showing that 100 nm-thick micropillars deform homogeneously in tension. Other studies [8,9,14–17] reported contradicting results with the formation of shear bands even for the smallest pillars. Some authors found an increase of yield strength for decreasing diameter [7,14,15], while others did not detect any change or even a small decrease [6,9,16–18]. Guo et al. [11] deformed Zr-based thin film metallic glasses (TFMGs) in tension inside a TEM, with 100 × 100 × 250 nm sized specimens sustaining plastic deformation up to 45% but without stress measurement. Deng et al. [10] and Jiang et al. [19] confirmed these results for 50 nm-thick Cu₄₉Zr₅₁ and Ni₆₀Nb₄₀ TFMGs reporting a ductility up to 12% and 40%, respectively, but the test was not instrumented for force measurement. Tian et al. [20], using dog-bone shape specimens, extracted the full stress-strain curve of 200 nm-thick Cu₄₉Zr₅₁ TFMGs showing large elastic deformation ~4% coupled with a yield strength close to the theoretical value of 3.7 GPa. Lastly, in our recent studies [21,22], we found size effects on the failure behavior and fracture toughness of submicrometer sized Zr₆₅Ni₃₅ TFMGs related to geometric confinement and not

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due to a compositional or structural change as a function of thickness.

A conclusive understanding of the origin of size effects and of the associated mechanisms for the formation or not of catastrophic shear bands is still missing. The difficulty comes from the challenge to generate consistent and reproducible data on extremely small specimens, while avoiding artifacts. In micropillar compression, the sample geometry can have a rounded tip and tapered shape, while imperfect punch-pillar contact leads to stress concentration [8]. Information about the magnitude of the stress in *in-situ* TEM is often missing, while the attainment of large plastic deformation is strongly dependent on the loading set-up, machine stiffness and specimen alignment affecting shear bands stability [2,10]. Specimens are most often produced by Focus Ion Beam (FIB) involving high energy Ga^+ ions. This operation alters the mechanical behavior by chemical softening and free-volume generation [23,24]. Lastly, a complete picture of mechanical size effects over a broad range of specimen geometries and sizes is missing, with most reported works involving only a limited number of test specimens.

The objectives of this work are to investigate the behavior of $\text{Zr}_{65}\text{Ni}_{35}$ (% at.) TFMGs deformed in tension and to analyze the size dependent strength and ductility. More specifically, we report the successful fabrication of freestanding fully amorphous $\text{Zr}_{65}\text{Ni}_{35}$ metallic glass tensile test specimens avoiding FIB milling, while studying a broad range of geometries to investigate how size affects strength and ductility. This lab-on-chip method [25,26] allows the testing of a large number of almost perfectly aligned micron and sub-micron sized elementary specimens, around one thousand in this study, each one giving one specific point on the stress-strain curve.

The outline of the paper is the following. The lab-on-chip mechanical testing method is presented in Section 2 providing all the details of the fabrication and the extraction of mechanical properties. Section 3 focuses on the results about mechanical size effects, SEM and high resolution TEM (HRTEM) observations. Lastly, Section 4 is dedicated to the discussion.

2. Experiments

2.1. Lab-on-chip technique for mechanical testing

The on-chip technique based on residual stress actuated microtensile testing structures developed at Université catholique de Louvain (UCL) [25–27] is applied here for the first time to metallic glasses (Fig. 1a and b). A long dogbone test specimen made by metallic glass overlaps a Si_3N_4 actuator beam involving tensile residual stress ~ 1 GPa [28] both patterned by lithography. The “actuator + specimen” constitutes one elementary test structure. Upon release (Fig. 1b), the actuator contracts and pulls on the specimen beam, which is almost perfectly aligned with the loading direction, until force equilibrium is reached [25,26]. Tests are simultaneously performed on hundreds of specimens with different geometries for the determination of the uniaxial entire stress-strain response up to fracture. We successively present hereafter (i) the fabrication steps involved in the generation of the test structures, (ii) the data reduction scheme to extract the stress and strain applied to each specimen, and (iii) the mechanical stability analysis for this specific test configuration.

2.1.1. Fabrication of micro-tensile freestanding specimens

A complete description of the generic steps for the production of the on-chip test structures can be found in Refs. [25–27]. Its applications to TFMGs is explained hereafter. Firstly, Si (100) wafer is cleaned with $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ solution followed by a 2% vol. $\text{HF}:\text{H}_2\text{O}$. The actuator layer (Si_3N_4) is deposited by Low Pressure Chemical

Vapor Deposition (LPCVD). The difference in thermal expansion coefficient between the thin Si_3N_4 layer and the Si substrate combined with the high deposition temperature (790 °C) leads to ~ 1 GPa residual stress in the actuator layer [26–28]. The actuator residual stress (σ_{ra}) has been measured using Stoney equation by evaluating the variation of the radius of curvature of a Si wafer coated with a Si_3N_4 layer, see Ref. [28].

Then, the actuator mismatch strain (ϵ_a^{mis}) – which is an essential ingredient for the determination of the stress applied to the specimen (see further) – is obtained as $\frac{\sigma_{ra}}{E_a}(1 - \nu_a)$ from equibiaxial elastic conditions, where E_a – measured using nanoindentation – is equal to 250 GPa and ν_a has been taken equal to 0.24 [25–28]. In addition, ϵ_a^{mis} has also been directly estimated from measurements of the contraction of free actuators upon release [27,28]. For all thicknesses investigated ϵ_a^{mis} was found equal to -0.003 ± 0.0005 in agreement with earlier results, see Refs. [25–27]. The Si_3N_4 layer is then patterned in the shape of long beams (see Fig. 1a and b) by photolithography and by dry etching (SF_6 plasma). The thickness of the actuator layer is modified for each specific specimen thickness in order to optimize the accuracy of the method (see Ref. [26] for details about the error analysis). Here the actuator thicknesses are equal to 80, 100, 160 or 350 nm for specimen thicknesses equal to 110, 200, 360 and 550 nm, respectively. The length of the actuator beam is varied between 100 and 4000 μm in order to generate different levels of deformation in the test specimens. The actuator beam width is equal to 15 μm . The specimen layer is deposited by DC-magnetron sputtering (details in Refs. [21,22]). It is patterned using a photolithography lift-off technique.

The specimens have a dog-bone shape as sketched in Fig. 1a and b in order to ensure perfect uniaxial tension condition along the entire section and to avoid stress concentration at the edges. A variation of the specimen thickness along the width has been observed on SEM cross-sections, caused by the lift-off process. The impact of this non-uniform thickness on the extracted mechanical properties is discussed in the Appendix. The residual stress in the specimen has been measured using Stoney equation by evaluating the difference of the curvature radius of a Si wafer coated with the $\text{Zr}_{65}\text{Ni}_{35}$ layer [25–27]. Then, the specimen mismatch strain (ϵ^{mis}) is given by $\frac{\sigma_r}{E}(1 - \nu)$ where E and ν have been extracted using Brillouin spectroscopy as equal to 72 GPa and 0.39, respectively (see Ref. [22]). For all thicknesses, ϵ^{mis} was found equal to $+0.0017 \pm 0.0003$, where the positive sign indicates that the stresses are compressive (i.e. the corresponding residual stress results from an elastic strain that is exactly equal to $-\epsilon^{mis}$).

The release of the actuator is performed by wet etching the silicon substrate in 10% vol. TMAH (tetramethylammonium hydroxide): H_2O solution at 85 °C. Freestanding films have been obtained after 17 min immersion, a condition which gives a minimum underetching equal to ~ 20 μm . After release, the actuator and the specimen beams are then detached from the substrate and thus free to move. The force relaxes in the actuator beam while it increases in the specimen beam until equilibrium is reached [25–27], see SEM images in Fig. 1c and d. The specimen beam elongates and this gives rise to a displacement between the fixed and moving cursors (Fig. 1c). After etching, the samples are left in H_2O first and then in isopropanol for 50 min in order to stop the etching reaction. Critical point drying is then performed in order to avoid stiction of the structures onto the underlying substrate.

All the specimen dimensions are summarized in Table 1: these dimensions are significantly larger than in the works cited earlier [6,7,10,11,14,20] covering almost one and even two decades in terms of specimen cross-section area and volume variations, respectively.

2.1.2. Extraction of stress and strain

The stress (σ) and mechanical strain (ϵ^{mech}) can be directly

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